

# DESIGN, FABRICATION AND TESTING OF A DUAL CATALYST AMMONIA REMOVAL SYSTEM FOR A URINE VCD UNIT

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by

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Prepared Under Contract No. NAS2-10237

by

**GATX**

**GARD, INC.**

7449 NORTH NATCHEZ AVENUE  
NILES, ILLINOIS 60648

for



AMES RESEARCH CENTER  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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## FOREWORD

The investigation described herein was conducted by GARD, INC/GATX during the period of May, 1979 through June, 1980 under NASA Contract NAS2-10237. The Project Engineer was P. Budininkas who was assisted by R.J. Honegger and E.K. Krug. The Technical Monitor was M.I. Leban, NASA Ames Research Center, Moffett Field, California.

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## SUMMARY

A three-man capacity catalytic system for the recovery of water from urine was designed, constructed, and tested. The system was designed to operate with feed streams containing high concentrations of urine vapor and only 5-7% of oxygen (supplied either as air or elemental oxygen) for the oxidation of ammonia and volatile organic vapor. The system can operate either in a flow-through or a recycle mode and is capable of accepting urine vapor produced by a VCD evaporator.

The testing program consisted of short preliminary and optimization tests, an endurance test consisting of 74 hours of continuous operation, and recycle tests using both air and oxygen. The system was designed for a urine processing rate of 0.86 l/hr; however, it was tested at rates up to 1.2 l/hr. The system was tested with untreated urine which was evaporated by an electrically heated evaporator.

The quality of the recovered water meets the U.S. Drinking Water Standards, with the exception of a low pH. Because of urea decomposition followed by the removal of ammonia in the catalytic reactor, the accumulation of solids in the urine sludge is reduced to approximately 65% of the anticipated value.

## INTRODUCTION

Previous investigations (NASA CR 151930 and NASA CR 152227) have demonstrated the feasibility of water recovery from untreated urine by the catalytic treatment of its vapor for the removal of ammonia and volatile organic carbon. Testing of a dual catalyst system integrated with a wick evaporator (NASA CR 152227) demonstrated its capability on a 4-man rated scale; however, the capacity of the integrated system was limited by the rate of urine vapor generation by the wick-evaporator. Since the wick-evaporator produces an air-vapor stream containing only 3-5 volume % of urine vapor, large excess of air is required to achieve a 4-man rating. Consequently, the size of the dual catalyst system and the associated volume, weight and power penalties become increased.

Bench scale tests indicated that the dual catalyst system is capable of removing ammonia and volatile organic carbon from feed streams containing up to 95% of urine vapor and only approximately 5-7% of oxygen, supplied either as air or elemental  $O_2$ . The use of feed containing high vapor concentrations minimizes the volume, weight, and power requirements of the dual catalyst system.

Certain urine evaporation schemes, notably vapor compression distillation (VCD), are capable of producing high concentrations of urine vapor combined with an effective recovery of the latent heat of evaporation. At the present time, the need to minimize the formation of ammonia due to urea decomposition limits the temperature of urine evaporation and necessitates chemical pretreatment of urine. An integration of an



evaporator such as vapor compression distillation unit with the dual catalyst system would result in an improved quality of the recovered water. Since ammonia would be removed from urine vapors catalytically, no pretreatment of urine would be required and evaporation could be performed at higher temperatures, resulting in a higher capacity of the system. In addition, the volatile organics would be completely removed from urine vapor by the catalytic oxidation into innocuous products.

The objective of this investigation was to design, fabricate, and test a dual catalyst system capable of treating feeds consisting of high concentrations of untreated urine vapor mixed with minimal concentrations of oxygen or air. Vapor of untreated urine was to be produced by thermal evaporation to simulate urine vapor produced by a three-man rated vapor compression distillation unit.

To achieve the objectives of this investigation, the program consisted of the following tasks:

1. Catalyst Optimization.- Bench scale tests were performed on ammonia oxydation catalyst employed previously (NASA CR 152227) to establish data required for catalyst bed sizing and design of a 3-man rated system for feeds containing high urine vapor concentrations.

2. Design and Fabrication of the Catalytic System.- A catalytic system adequate for treating the urine of a three-man crew was designed. The system is capable of operating in either a flow-through or a recycle mode. The catalytic system was fabricated from commercially available components.

3. System Testing.- The system was tested using untreated urine.

The testing program consisted of the following:

- a. Preliminary tests,
- b. Tests to demonstrate the performance of the system under selected operational parameters,
- c. Endurance test for continuous operation,
- d. Recycling of noncondensable gases.

The quality of the recovered water and the composition of the vent gases were determined by appropriate analytical procedures.

## BENCH SCALE TESTS

### Catalyst Optimization

To establish data for the catalyst bed sizing and design and to determine the operational parameters, bench scale optimization tests were performed on the ammonia oxidation catalyst. Because the concentration of urine vapor in feed stream has no direct effect on the  $N_2O$  decomposition, data from previous investigation (NASA CR-152227) were used for sizing of  $N_2O$  decomposition reactor.

The basic requirements for a satisfactory performance of the catalyst are:

- a. complete removal of  $NH_3$  from the vapor stream,
- b. no  $NO_x$  formation.

The following variables were tested:

- a. Catalyst temperature,
- b.  $O_2$  concentration in feed (supplied either as elemental  $O_2$  or air),
- c. Space velocities,
- d. Effect of  $N_2O$  presence in feed.

All tests were performed in a flow-through experimental arrangement depicted schematically in Figure 1, consisting of an electrically heated evaporator, heated catalytic reactor, gas supply and mixing lines, appropriate flowmeters and temperature controls, a water cooled condenser, and a condensate collection vessel. The Pyrex glass catalytic reactor, 27 mm I.D., was surrounded by a tubular electric heater which provided an adequate regulation of the catalyst bed temperature. The catalyst bed containing 50 cc of platinum type catalyst was placed on top of a 50 cc heated bed of 3.2 mm alumina pellets. The feed mixture passing

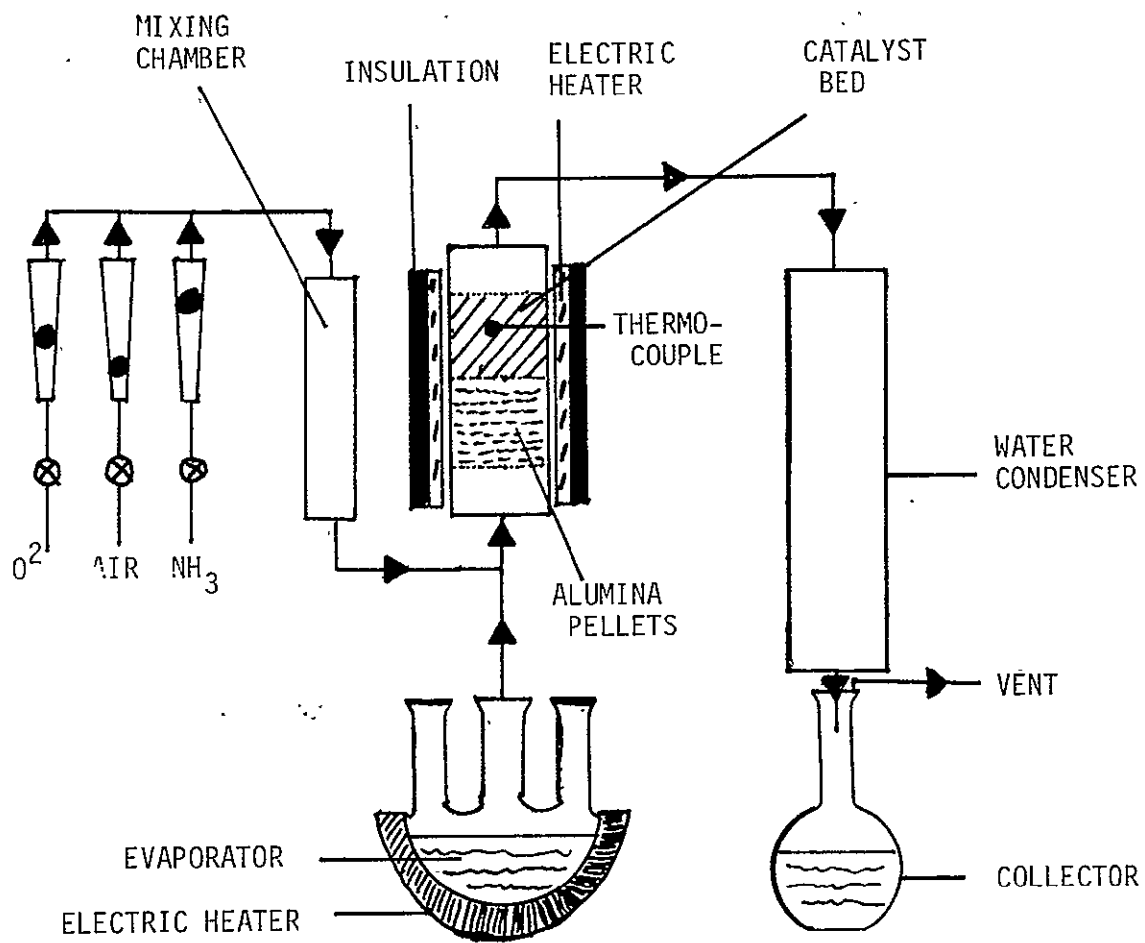


Figure 1. CATALYST TESTING SYSTEM

through these inert alumina pellets becomes preheated to the reaction temperature before entering the catalyst bed. This arrangement resulted in an essentially constant temperature through the entire catalyst bed.

#### Tests with Water Vapor-Ammonia Mixtures

Because evaporation of urine produces vapor containing variable concentrations of ammonia, the initial optimization tests were performed by vaporizing water and adding appropriate concentrations of ammonia gas to the vapor stream. Metered amounts of oxygen and air (or nitrogen) were also added to the vapor stream before entering the catalytic reactor. By regulating the gas flows and water evaporation rate, mixtures of desired composition could be obtained at selected feed rates.

The results of these tests are summarized in Table 1 and indicate that a complete removal of  $\text{NH}_3$  requires the following operational parameters:

- a. Catalyst temperature above  $200^\circ\text{C}$ ,
- b. Space velocities up to  $10,400 \text{ hr}^{-1}$ ,
- c. Oxygen minimum concentration in feed mixture 0.5%.

#### Tests with Untreated Urine

In addition to ammonia, vapor of untreated urine contains variable concentrations of volatile hydrocarbons and carbon dioxide; therefore, tests with artificial mixtures cannot completely represent the actual feed composition. For this reason, bench scale tests were performed using untreated urine vapor and admixing oxygen either as air or elemental  $\text{O}_2$ . The results of these tests are summarized in Table 2.

TABLE 1. BENCH SCALE OPTIMIZATION TESTS WITH  
ARTIFICIAL GAS MIXTURES

Feed Composition			Temperature, °C	Space Velocity, hr <sup>-1</sup>	NH <sub>3</sub> in Condensate mg/l
Water Vapor, %	O <sub>2</sub> , %	NH <sub>3</sub> , ppm			
<u>Air</u>					
60.6	8	1,000	250	3,300	ND
75.5	4	1,000	250	2,500	ND
93.6	1	1,000	250	2,100	ND
95.7	0.5	1,000	250	2,100	ND
96.9	0.25	1,000	250	2,100	0.09
96.9	0.25	1,000	250	2,100	0.03
97.5	0.10	1,000	250	2,100	0.03
93.6	1	1,000	200	2,100	28.6
95.7	0.5	1,000	225	2,100	ND
95.6	0.5	1,000	200	2,100	0.5
79.5	4	1,000	200	2,500	2.5
81.1	4	1,000	250	4,900	ND
95.7	0.5	1,000	250	4,200	ND
95.7	0.5	1,000	250	4,200	ND
95.7	0.5	1,000	250	6,300	ND
95.7	0.5	1,000	250	6,300	ND
95.7	0.5	1,000	250	8,400	ND
95.7	0.5	1,000	250	10,400	ND
<u>Oxygen</u>					
98.0	0.05	1,000	250	10,200	228
98.0	0.10	1,000	250	10,200	4
97.8	0.25	1,000	250	10,200	ND
97.8	0.25	1,000	225	10,200	ND
97.8	0.25	1,000	200	10,200	ND
97.8	0.25	1,000	175	10,200	0.9

ND = Not Detectable

TABLE 2. TESTS WITH VAPOR OF UNTREATED URINE

Feed		Temperature, °C	Space Velocity, hr <sup>-1</sup>	Condensate Analysis	
Urine Vapor, %	O <sub>2</sub> , %			NH <sub>3</sub> , mg/l	T.O.C., mg/l
<u>Air</u>					
95.2	1.0	250	4,200	ND	0.8
97.6	0.5	250	4,100	820	10.9
95.2	1.0	225	4,200	7.5	4.1
95.2	1.0	250	10,500	ND	0.8
<u>Oxygen</u>					
99.5	0.5	200	4,000	3.9	55
97.8	2.2	200	4,100	781	117
99.0	1.0	225	4,100	1.5	4.7
97.8	2.2	250	4,100	ND	2.1
99.0	1.0	250	4,100	ND	2.1
99.5	0.5	250	4,000	3.5	5.0
99.5	0.5	250	4,000	5080	4.9
99.0	1.0	250	10,000	ND	1.6

ND = Not Detectable

From the obtained data, the following conclusions can be made:

- a. A catalyst temperature of  $250^{\circ}\text{C}$  is required to achieve a complete oxidation of ammonia and volatile organic carbon,
- b. At least one percent (by volume) of oxygen (as air or elemental  $\text{O}_2$ ) is necessary to completely remove ammonia; however, the removal of organic carbon will require a higher  $\text{O}_2$  concentration,
- c. Under these conditions, the catalyst is effective up to space velocities of  $10,000 \text{ hr}^{-1}$ .

Anticipating the possibility of a recycle operation during which the recycling gases may contain high concentrations of nitrous oxide, tests were performed to determine the effect of  $\text{N}_2\text{O}$  concentration on the removal of  $\text{NH}_3$  and volatile organic carbon from untreated urine vapor. All tests were performed under identical conditions, i.e., catalyst temperature of  $250^{\circ}\text{C}$ , urine evaporation rate of  $2 \text{ cc/min}$ , and oxygen concentration 3.2-3.1% (supplied as air). Nitrous oxide was admixed to the feed in various concentrations, and the concentrations of ammonia and total organic carbon (TOC) in the product condensate were determined. The results of these tests are summarized in Table 3. These tests indicate that the presence of 2.5% or higher concentrations of  $\text{N}_2\text{O}$  in feed will have an effect on the performance of the catalyst and may cause incomplete removal of ammonia and organic carbon.



TABLE 3. EFFECT OF NITROUS OXIDE

Temperature 250°C  
Space Velocity 3800 hr<sup>-1</sup>

Feed Composition			Analysis of Condensate		
Urine Vapor, %	O <sub>2</sub> (as air), %	N <sub>2</sub> O, %	pH	NH <sub>3</sub> , mg/l	TOC, mg/l
85	3.2	0	4.25	ND	ND
85	3.2	0	3.38	ND	0.8
85	3.2	0.13	4.42	ND	ND
85	3.2	0.13	4.14	ND	ND
85	3.2	0.25	4.50	0.2	ND
83	3.1	2.5	4.50	0.2	ND
83	3.1	2.5	5.54	0.8	2.9
82	3.1	3.1	6.68	12.7	

ND = Not Detectable

## DESIGN AND FABRICATION OF A THREE-MAN SYSTEM

### Design Requirements

The catalytic system must be adequate for treating not only the urine output of a 3-man crew but, in addition, process urinal flush water and shower and laundry concentrated brine. A recent report<sup>(1)</sup> on VCD developments fails to indicate the daily processing rate; however, it lists the feed stream composition in the proportions of 260 cc urine, 150 cc urine flush water and 410 cc wash water brine. Assuming a maximum urine production of 1,820 cc/man-day and the above proportions, the required total processing rate of a 3-man system becomes 17.22 liters/day. On this basis and the assumption that the water recovery system will operate 20 hrs/day, the nominal processing rate of the system will be 14.4 cc/min. .

The overall schematic of a catalytic urine treatment system is presented in Figure 2. The solid lines indicate operation in a flow-through mode; the broken line depicts return of the noncondensable gas when the system is operated in the recycle mode.

System design requirements and conditions based on the above considerations and catalyst operational parameters established by bench scale testing are summarized in Table 4. The calculated sizes and geometries of the basic system components are presented in Table 5.

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(1) LMSC - D626041, "Development of a Preprototype Vapor Compression Distillation Water Recovery Subsystem," November, 1978, Lockheed Missiles and Space Co.

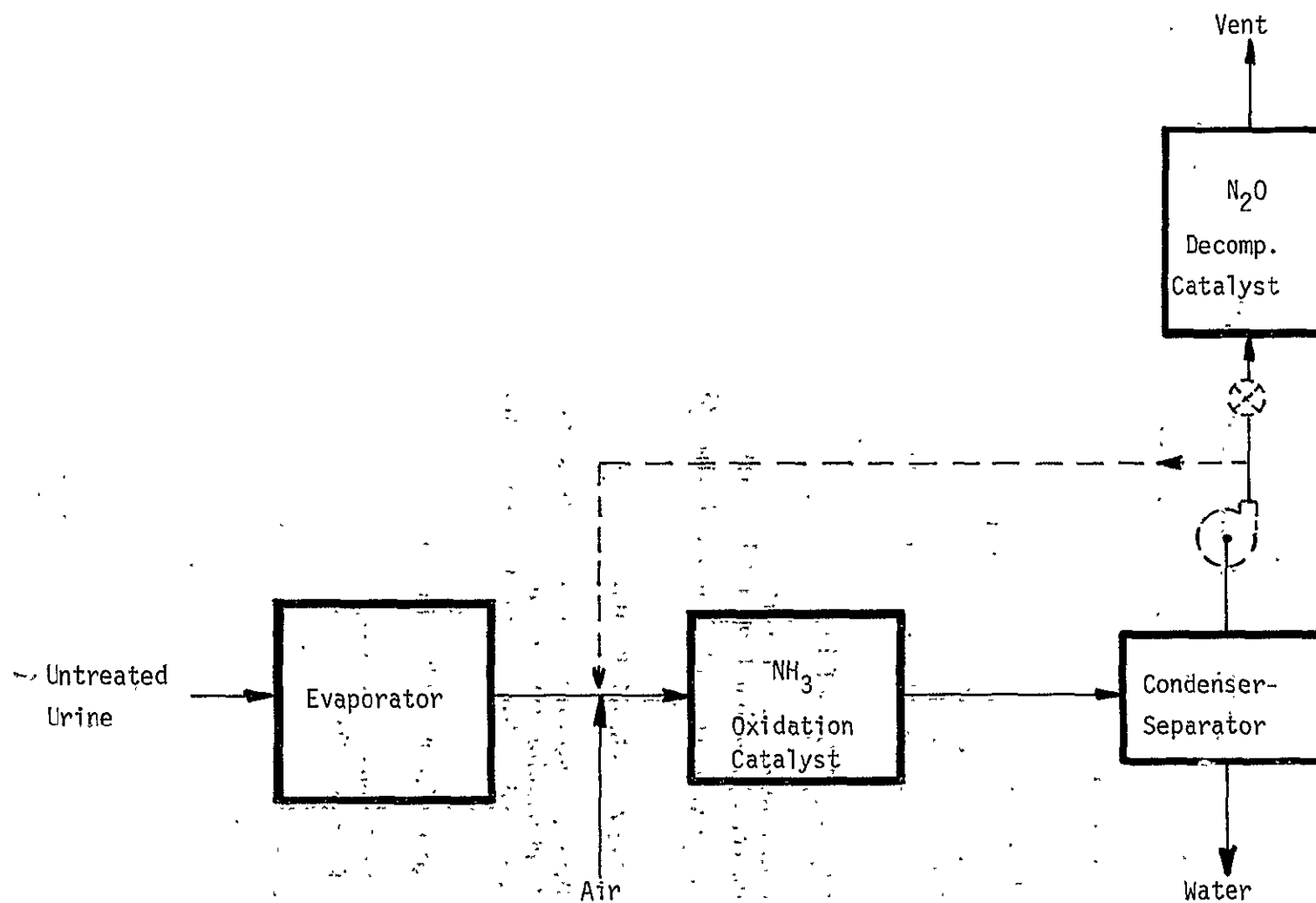


Figure 2. SCHEMATIC OF CATALYTIC SYSTEM FOR WATER RECOVERY FROM URINE

TABLE 4  
DUAL CATALYST SYSTEM DESIGN CONDITIONS

1. PROCESSING RATE		
Urine		1820 cc/man-day
Flush water		1050 cc/man-day
Urine & flush water -- 3 men		8610 cc/day
Wash water brine -- 3 men		8610 cc/day
Daily rate -- 3 men		17220 cc/day
Ave. rate for 20 hr/day operation		14.4 cc/min
2. NH <sub>3</sub> OXIDATION CATALYST		
Operational temperature		250°C
O <sub>2</sub> Concentration (supplied as air)		4%
Space velocity (ideal gas @ 1 atm, 25°C)		8000 hr <sup>-1</sup>
3. CONDENSATION/SEPARATION		
Mixture entering condenser (ideal gas, 25°C)		81% vapor, 19% air
Condensation temperature		16°C
Non-condensables/vapor leaving separator		2% vapor, 98% air
4. N <sub>2</sub> O DECOMPOSITION CATALYST		
Operational temperature		450°C
Space velocity		5600 hr <sup>-1</sup>

TABLE 5  
DESIGN SIZES AND GEOMETRY OF SYSTEM COMPONENTS

1. $\text{NH}_3$ OXIDATION BED		
Volume of catalyst		200 cc
Bed diameter		4.6 cm
Bed depth		12 cm
$\Delta p$ across bed @ $250^\circ\text{C}$		1.3 cm $\text{H}_2\text{O}$
2. $\text{N}_2\text{O}$ DECOMPOSITION BED		
Volume of catalyst		50 cc
Bed diameter		2.7 cm
Bed depth		8.7 cm
$\Delta p$ across bed @ $450^\circ\text{C}$		0.9 cm $\text{H}_2\text{O}$
3. CONNECTING TUBING		
Diameter		1.3 cm ( $\frac{1}{2}$ " )

## Fabrication

The main components of the system are the two catalytic reactors, the water cooled vapor condenser, and the recovered water collector, all interconnected by stainless steel tubing. Both catalytic reactors and the condenser were constructed from stainless steel; the water collector was a wide mouth glass bottle with a drain for the removal of the recovered water. All the components of the system were fabricated from commercially available materials and components.

Figure 3 shows the construction features of the  $\text{NH}_3$  oxidation catalytic reactors. Except for its size, the nitrous oxide decomposition reactor has the same configuration. The following designed construction and operational features are common for both reactors:

- a. The operational temperature is achieved by preheating the gas-vapor stream which then heats the catalytic granules,
- b. Heat is supplied by electric cartridge elements inserted into machined heating surfaces below the catalyst bed,
- c. Three thermocouple wells located at the inlet face, mid-point, and the outlet face of the catalyst bed are provided for the controlling and measuring thermocouples,
- d. Each reactor is sized to contain the nominal volume of the catalyst bed plus a reserve space equal to 50% of the nominal volume,
- e. Stainless steel mesh plugs are located below and above the heating elements to promote mixing and heating of feed mixture,

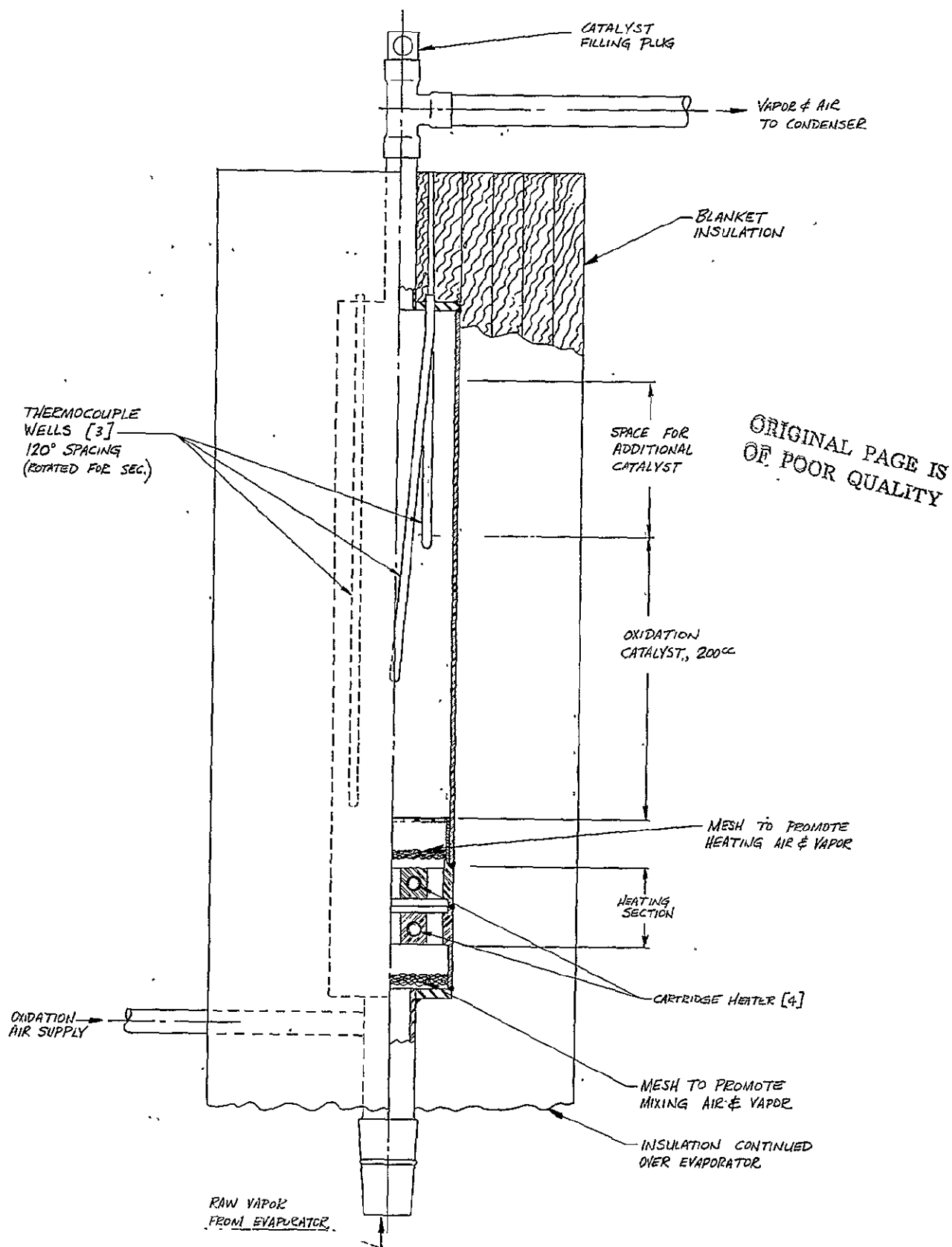


FIGURE 3.  $\text{NH}_3$  OXIDATION REACTOR ASSEMBLY

f. Reactors are covered with wrap-around thermal insulation.

Both catalytic reactors are shown in Figure 4. The  $\text{NH}_3$  oxidation reactor is heated by four (4) 62-Watt cartridge electric heaters, and has a 24/40 stainless steel tapered joint for integration with the glass evaporator; the  $\text{N}_2\text{O}$  decomposition reactor has two (2) 62-Watt cartridge heaters.

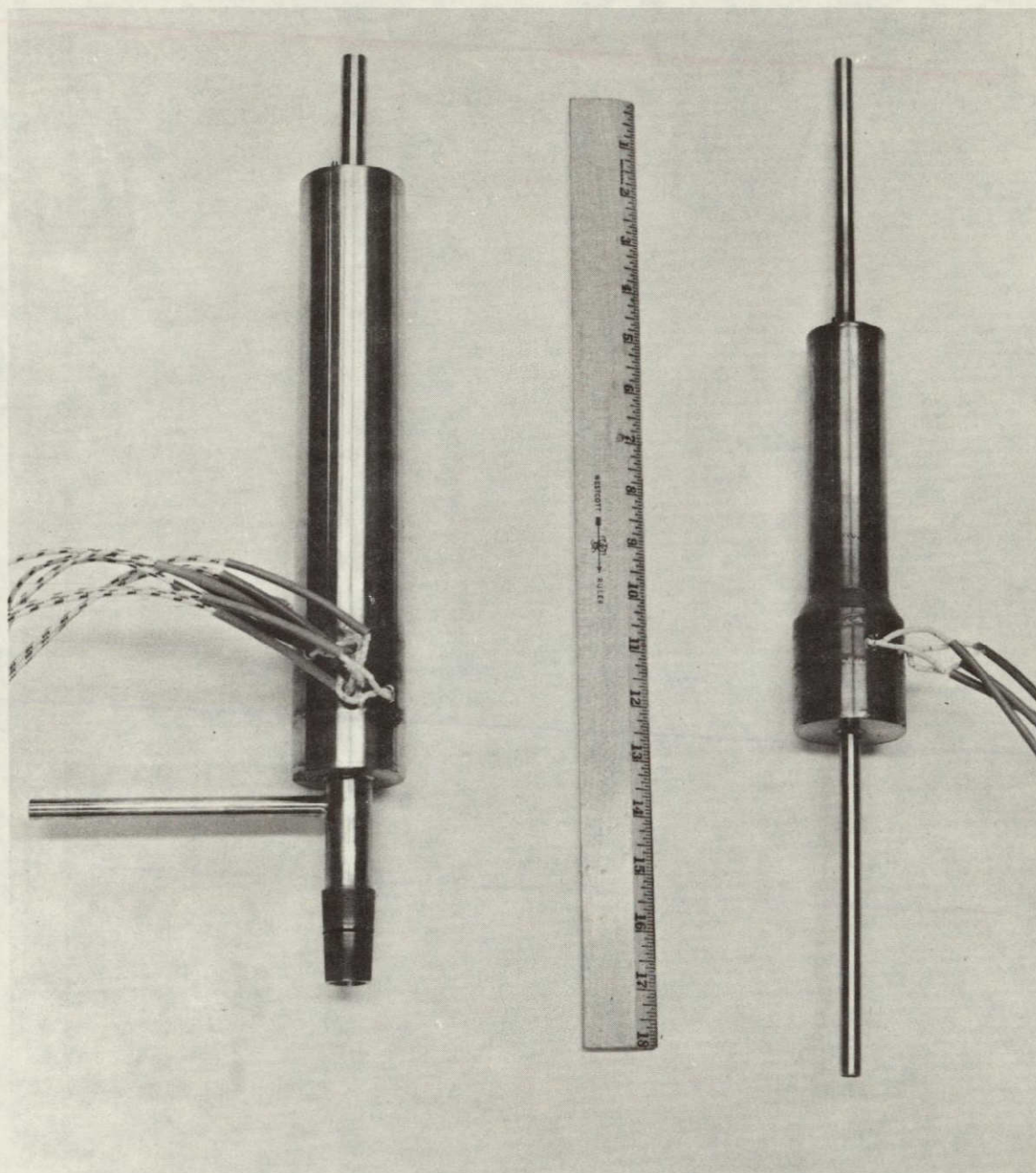
The  $\text{NH}_3$  oxidation reactor contains 201 g (220 cc) of 0.5 % Pt on 3.1 mm alumina pellets catalyst creating a 12 cm deep bed; the  $\text{N}_2\text{O}$  decomposition reactor contains 50 g (50 cc) of 0.5% Ru on 3.1 mm alumina pellet catalyst creating a 8.7 cm deep bed.

A stainless steel water cooled condenser with a nominal 10,000  $\text{cm}^2$  heat exchange area used in a previous program (contract NAS 2-9715) was initially installed into the system; however, it was later replaced by a glass condenser of adequate size for condensing the recovered water produced at the average processing rate of 14.4 cc/min.

### Three-Man System

The overall view of the three-man system is shown in Figure 5. The  $\text{NH}_3$  oxidation reactor is integrated with an electrically heated urine evaporator whose output is controlled by regulating the power input to its heaters. Untreated urine is pumped by a diaphragm pump from a reservoir to the urine evaporator at a rate adequate for maintaining a constant liquid level in the evaporator. A metered stream of oxidant gas (either air or oxygen) required to maintain the desired oxygen concentration in feed is added to the urine vapor before entering





$\text{NH}_3$  Oxidation Reactor-

$\text{N}_2\text{O}$  Decomposition Reactor

Figure 4. CATALYTIC REACTORS



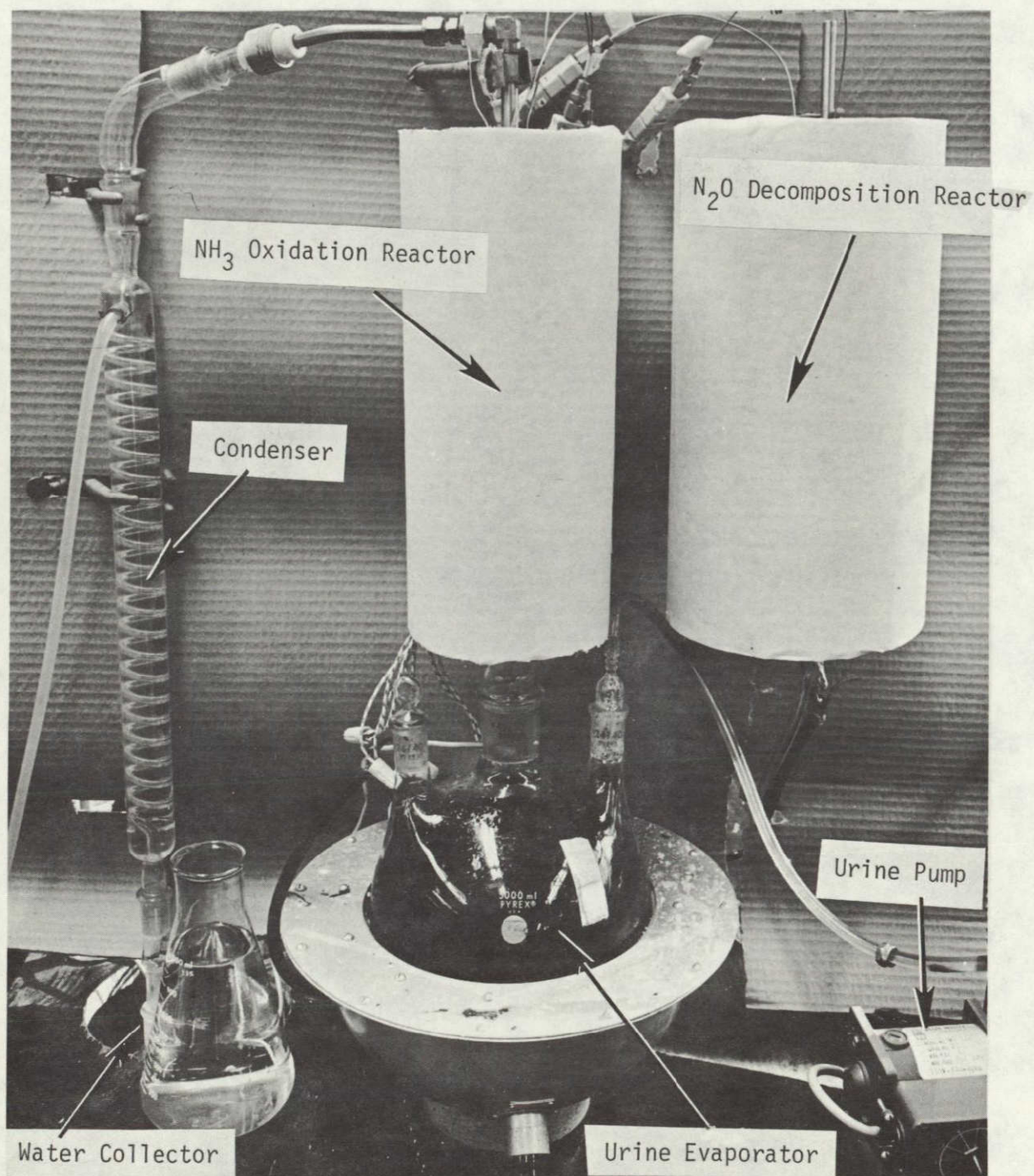


Figure 5. CATALYTIC WATER RECOVERY SYSTEM

the catalytic reactor. Ammonia and volatile organic compounds are oxidized in the  $\text{NH}_3$  oxidation reactor; the treated vapor, now free of  $\text{NH}_3$  and organic carbon, is condensed and collected in the water collector. The noncondensable gases are passed through the  $\text{N}_2\text{O}$  decomposition catalyst and vented.

When operating in the recycle mode, a gas blower located between the water collector and the  $\text{N}_2\text{O}$  decomposition catalyst recycles the noncondensable gas to the inlet of the  $\text{NH}_3$  oxidation reactor. Oxidant gas is added to the feed stream for maintaining a desired oxygen concentration, while an equivalent amount of noncondensable gas is vented through the  $\text{N}_2\text{O}$  decomposition reactor.

### Functional Testing

Functional testing was performed to establish the startup and operational procedures and to compare the observed levels of parameters with the design values. The startup procedures involve passing air through the system while preheating both reactors to their operational temperatures. When the temperatures of the reactors stabilize, the heaters of the urine evaporator are energized. After attaining the preset temperature of the evaporator, the flow of the oxidant is adjusted to an appropriate level. To avoid transfer of untreated urine to the water collector, it is important that the  $\text{NH}_3$  oxidation reactor becomes fully preheated before the preheating of the evaporator is initiated.

Initial testing indicated a large variation of temperatures across the catalyst bed of the  $\text{N}_2\text{O}$  decomposition reactor. This was attributed

to an insufficient heat transfer by the relatively low flow of gas through the reactor. This problem was solved by installing a pair of four-inch . long tubular clamp-on heaters around the reactor and using them to control the reactor temperature.

## SYSTEM TESTING

The system testing program consisted of the following test series:

1. Performance tests to determine the effects of process variables on the quality of the recovered water,
2. Testing under reduced pressure,
3. Duration test, and
4. System operation in a recycle mode.

All tests were performed using untreated urine collected daily from male volunteers; however, Dow Corning H-10 antifoam agent was admixed at a rate of 0.25 cc per liter of urine when foaming problem was encountered at high evaporation rates. The catalysts indicated no decrease in activity through the entire testing program consisting of 260 hrs of actual operation.

Sampling and analyses were performed to obtain processing rate and material balance, to characterize feed and vent gases, and to determine the quality of the recovered water. Typically, the following analyzes were performed:

- a. Feed - selected samples of feed vapor were withdrawn and analyzed to determine the concentrations of  $\text{NH}_3$  and organic carbon,
- b. Recovered water - the quality of the recovered water was monitored by routinely testing for pH,  $\text{NH}_3$ , organic carbon (TOC), conductivity, turbidity, nitrate and nitrite,
- c. Vent gas - selected samples were analyzed for  $\text{NH}_3$ ,  $\text{NO}_x$ , and  $\text{N}_2\text{O}$ ,
- d. Recycling gas - concentrations of  $\text{O}_2$ ,  $\text{CO}_2$ , and  $\text{N}_2\text{O}$  were monitored.

## Performance Tests

These tests were performed to determine the effects of catalyst temperature, feed gas oxygen concentration (supplied either as air or elemental  $O_2$ ), and space velocity on the quality of the recovered water and to establish optimum operational parameters. During each test the system was brought to the selected conditions and operated under these conditions for several hours while collecting a sample of the recovered water for analysis.

The results of the performance tests are presented in Table 6. The effects of each variable are summarized below.

Temperature.- The minimum temperature required for a complete removal of  $NH_3$  is  $240^{\circ}C$ .

Oxygen Concentration in Feed.- When oxygen was supplied as air, maintaining its concentrations at 4% was sufficient to achieve the removal of  $NH_3$ ; however, when elemental oxygen was employed, a concentration of 12% was required. The probable cause of the difference between air and elemental oxygen is due to the lower flowrate of oxygen, causing insufficient mixing and channeling and resulting in insufficient oxygen levels in some areas of the ammonia oxidation reactor. This can be remedied by a reactor design modification, as was partially proven by a test in which oxygen was added directly to the evaporator for better mixing and resulted in improved results.

Space Velocity.- Tests were performed at space velocities ranging from  $5,673 \text{ hr}^{-1}$  to  $8,238 \text{ hr}^{-1}$ , representing evaporation rates of 11.3-16.4 cc/min. The quality of the water remained the same through this space velocity range.

As testing continued and fresh urine was continuously added to the evaporator, the solids concentration of the accumulating sludge progressively

TABLE 6. EFFECTS OF PROCESS VARIABLES ON THE QUALITY OF  
RECOVERED WATER

Test No.	Ammonia Catalyst Temp., °C	O <sub>2</sub> (as air), %	Space Velocity, hr <sup>-1</sup>	Recovered Water Analysis					
				pH	NH <sub>3</sub> , mg/l	T.O.C., mg/l	Conductivity, Micromhos	Turbidity, JTU	NO <sub>x</sub> , mg/l
<u>Catalyst Temperature</u>									
1	260	4	5031	4.92	ND	<2.0	8	0.06	0.02
2	240	4	5031	5.00	ND	<2.0	6	0.04	<0.02
3	240	4	5031	3.74	ND	<2.0	50	0.04	0.29
4	220	4	5031	6.16	3.5	5.0	23	0.01	0.15
5	230	4	5031	5.73	1.5	3.0	16	0.04	0.77
6	240	4	5031	4.54	ND	<2.0	14	0.05	-
<u>Oxygen Concentration in Feed</u>									
7	250	4	5031	4.54	ND	ND	14	0.05	0.15
8	250	3	4752	5.52	1.5	3.0	19	0.03	0.18
9	250	4	5031	4.46	ND	3.0	14	0.05	0.11
10	250	4	5031	4.53	ND	3.0	18	0.16	0.14
11	250	4	5031	4.30	ND	3.5	24	0.06	0.14
13	250	4	5031	4.37	ND				0.14
15	250	6	5703	4.38	ND	ND	25	0.04	0.15
23	250	6	5703	3.92	ND	3.0	48	0.06	0.16
24	250	6	5703	4.19	ND	ND	26	0.11	0.10

ND = Not Detectable

TABLE 6. EFFECTS OF PROCESS VARIABLES ON THE QUALITY OF  
RECOVERED WATER - Continued

Test No.	Ammonia Catalyst Temp., °C	O <sub>2</sub> (as air) %	Space Velocity hr <sup>-1</sup>	Recovered Water Analysis					
				pH	NH <sub>3</sub> , mg/l	T.O.C., mg/l	Conductivity, Micromhos	Turbidity, JTU	NO <sub>x</sub> , mg/l
<u>Oxygen Concentration in Feed</u>									
25	250	4	5703	4.05	ND	3.0	30	0.10	0.18
26	250	4	5031	4.19	ND	3.0	27	0.08	0.08
<u>Oxygen Concentration in Feed, Supplied as Elemental O<sub>2</sub></u>									
12	250	4	4254	8.93	596		2300		
14	250	4	4244	8.91					
16	250	6	4334	8.79	466	1800	7000	<0.01	ND
17	250	8	4428	7.28	144	80	800	0.01	0.20
18	250	10	4527	4.36	1.5	12	40	0.03	0.25
19	250	10	4527	3.88	4.4	16	90	0.04	0.25
20	250	12	4630	3.97	ND	5.0	40	0.04	0.18
21	250	12	4630	3.98	ND	19	44	0.06	
22	250	15	4793	4.19	ND	7	27	0.09	
<u>Space Velocity</u>									
30	250	4	5673	3.47	ND	ND	150	0.4	0.73
31	250	4	5928	3.62	ND	1.0	102	0.5	ND
32	250	4	6633	3.66	ND	ND	90	0.2	0.47
33	250	4	7533	3.78	ND	2.0	80	0.15	--
34	250	4	8238	3.89	ND	1.0	70	0.1	--



increased. Samples of urine vapor were withdrawn after different volumes of urine were treated and analyzed for  $\text{NH}_3$  and total organic carbon. The results of these analyzes indicate that the concentrations of both ammonia and carbon in vapor increase with the increase in sludge concentration in the evaporator.

#### Testing Under Reduced Pressure

Three (3) tests were performed to determine the effect of system operation under reduced pressure on the quality of recovered water. The system was maintained at a pressure of 540 mmHg and urine was evaporated at  $65\text{--}68^\circ\text{C}$ , providing a water recovery rate of 7.5 cc/min. The results of these tests are summarized in Table 7 and indicate that the system is capable of operating under reduced pressure.

#### Duration Test

A duration test was run for 74 consecutive hours at an average water recovery rate of 16.6 cc/min. The system was operated in a flow-through mode using air as oxygen source. Untreated urine was used through the entire testing period; however, foaming agent was added to minimize foaming in the evaporator. After startup, the system operated completely automatically and did not require any manual adjustments. The ammonia oxidation catalyst was maintained at  $250^\circ\text{C}$  and the  $\text{N}_2\text{O}$  decomposition catalyst at  $500^\circ\text{C}$ . Recovered water was condensed at  $15\text{--}16^\circ\text{C}$ .

Sampling and analyzes were performed periodically to characterize the feed vapor and to determine the quality of the recovered water. The results

TABLE 7. EFFECT OF REDUCED PRESSURE

Test No.	Pressure, mmHg	Temp. of Evap., °C	Space Velocity, hr <sup>-1</sup>	Analysis of Recovered Water					
				pH	NH <sub>3</sub> , mg/l	T.O.C., mg/l	Conductivity, μmhos	Turbidity, JTU	NO <sub>x</sub> , mg/l
27	540		5031	4.47	<0.1	<1	43	0.2	ND
28	540	68	5031	4.52	<0.1	<1	17	0.2	
29	540	65	5031	4.31	<0.1	<1	24	0.4	ND

ND = Non detectable

of these tests are presented in Table 8. As the concentration of urine solids in the evaporator increases, progressively higher concentrations of  $\text{NH}_3$  appear in the feed vapor causing an increased oxygen demand; however, the concentrations of organic carbon remain essentially constant. After thirty hours of operation, an increase in oxygen concentration from 4% to 6% was required to produce water containing no  $\text{NH}_3$ .

The combined volume of water recovered during the entire duration test was analyzed for pH,  $\text{NH}_3$ , TOC, conductivity, turbidity, nitrate, and nitrite, and metals. The results are presented in Table 9 and compared with the U.S. Drinking Water Standards.

The recovered water meets all the U.S. Drinking Water Standards with the exception of the low pH value. Since the recovered water is unbuffered, very low concentrations of acidic components produced during the oxidation process effect a rather large change in the pH value.

The material balance for the entire duration, run is presented in Table 10. It indicates that the total solids remaining in the sludge constitute only 64.5% of the solids anticipated from 82 liters of urine. The main loss of solids occurred because of urea decomposition, followed by the removal of ammonia produced in the catalytic reactor. In addition, some volatile organic compounds were vaporized and removed by oxidation to carbon dioxide.

TABLE 8. DURATION TEST

Date	Time	Feed Vapor				Recovered Water				
		O <sub>2</sub> (as air), %	NH <sub>3</sub> , ppm	T.O.C., mg/l	pH	NH <sub>3</sub> , mg/l	T.O.C., mg/l	Conductivity, μmhos	Turbidity, JTU	NO <sub>x</sub> , mg/l
4-29	13:00	4								
	18:00	4								
	21:25	4								
4-30	0:10	4			4.52	ND	ND	26	0.2	Trace
	9:10	4	2,791	1,200	4.54	ND	ND	19	0.3	Trace
	11:00	4								
	14:00	4	3,356	1,300						
5-1	23:50	4			4.57	ND	ND	20	0.3	Trace
	8:20	4			6.25	6.0	4.0	41	0.3	
	9:30	5	1,639	1,250						
	11:15	4.5								
	12:00	5	4,836	1,350	6.5	6.0	12.0	51	0.1	
	15:00	5.5								
	18:30	6			4.19	ND	ND	28	0.3	
	22:45	6								
5-2	0:40	6			4.25	0.08	ND	28	0.3	No
	10:15	6			4.16	ND	ND	34	0.4	
	15:00	6	6,893	2,000	4.14	ND	5.0	42	0.3	

ND = Non Detectable

TABLE 9. QUALITY OF RECOVERED WATER

Parameter	Units	U.S. Drinking Water Standards		Recovered Water
		Recommended	Federal Mandated Limit	
pH	pH	6.5-8.5		4.63
Conductivity	µmhos			22
Turbidity	JTU	1-5		0.3
Ammonia	mg/l			ND
T.O.C.	mg/l			ND
Arsenic	mg/l	0.01	0.05	<0.0005
Barium	mg/l		1.0	<1.0
Cadmium	mg/l		0.01	<0.05
Chloride	mg/l	250		
Chromium	mg/l		0.05	<0.03
Copper	mg/l	1.0		
Iron	mg/l	0.3		<0.10
Lead	mg/l		0.05	<0.25*
Manganese	mg/l	0.05		
Nitrate	mg/l	45	45	
Silver	mg/l		0.05	<0.08*
Zinc	mg/l	5		
Mercury	mg/l		0.0002	0.0006
Total Dissolved Solids	mg/l	500		2.0

\* Detection Limits: Silver = 0.08 mg/l, Lead = 0.25 mg/l.

ND = Non Detectable.

TABLE 10. MATERIAL BALANCE

Total Urine Feed	82.0 l
Recovered water	73.9 l
Sludge in evaporator	7.0 l
Sampling of vapor	0.5 l
Total Measured Output	81.4 l
Sludge Analysis	
Density	1.128 g/cm <sup>3</sup>
Total solids	1,958 g
Inorganic salts	1,003 g
Urea	350 g
Other solids, organic	605 g
Supplied with Feed	
Total solids	3,038 g
Inorganic salts	1,161 g
Urea	1,098 g
Other solids, organic	779 g

### Recycle Tests

The system was converted to recycle operation by installing a recycle blower between the water collector and the  $N_2O$  decomposition reactor. The noncondensable gas was continuously recycled and returned to the feed stream at the inlet to the  $NH_3$  oxidation reactor. At the same point enough air or oxygen was added to maintain the desired  $O_2$  concentration. A slightly positive pressure of the recycling stream was maintained constant by venting the excess of noncondensable gas through the  $N_2O$  decomposition catalyst for the removal of  $N_2O$ .

The results of recycling tests with air are presented in Table 11 and with oxygen in Table 12. In both cases, the recovered water had essentially the same quality as previously observed with the flow-through operation. The vent gas contained, as anticipated, rather high concentrations of  $CO_2$ ; however, the vent gas did not contain any  $NH_3$ ,  $N_2O$ , or  $NO_x$ . These tests were started with a fresh batch of urine in the evaporator. As testing progressed, the sludge concentration increased and released larger than normal concentrations of  $NH_3$  and volatile carbon during startups, resulting in a marked decrease of oxygen concentration and an incomplete removal of  $NH_3$ ; however, these problems disappear after some time of operation.

The recycle mode produces recovered water and gas vent of the same quality as the flow-through mode with much lower oxidant feed rates and smaller  $N_2O$  decomposition reactor; therefore, it can be regarded as the preferred mode of operation.

TABLE 11. RECYCLING WITH AIR

Processing Rate: 16.7 cc/min  
 Air Feed Rate: 1.0 l/min  
 Gas Recycle Rate: 6.8 l/min

Date	Time	Feed to Reactor			Vent Gas:				Recovered Water					
		O <sub>2</sub> ,%	CO <sub>2</sub> ,%	N <sub>2</sub> O,ppm	CO <sub>2</sub> ,%	N <sub>2</sub> O,ppm	NH <sub>3</sub> ,ppm	NO <sub>x</sub> ,ppm	pH	NH <sub>3</sub> , mg/l	T.O.C., mg/l	Cond., µmhos	Turbidity, JTU	NO <sub>x</sub> , mg/l
5-9	13:30	4.4	0.26	641	12.8	Non-Detectable	Non-Detectable	Non-Detectable	4.54	ND	ND			
	14:50	4.4	0.26	697	10.7				4.44	ND	ND	23	0.1	ND
	15:30	4.4	0.32	611	17.7				4.54	ND	ND	12	0.1	ND
5-12	14:00	4.5	0.26	324	14.3				4.35	ND	ND	25	0.2	ND
	15:10	4.4	0.26	549	15.3				4.50	ND	ND	17	0.1	ND
	16:00		0.35	596					4.50	ND	ND	15	0.1	ND
5-13	11:00	4.2	0.23	746	12.6				4.26	ND	ND	25	0.2	ND
	12:00		0.32	487					4.49	ND	ND	16	0.1	ND
	13:00	4.3	0.38	489	18.9				4.55	ND	ND	15	0.1	ND
	14:00	4.2	0.48	631					4.64	ND	ND	12	0.1	ND
	15:30	4.3	0.49	647	25.9				4.62	ND	1.0	12	0.1	Trace
5-14	12:00	2.1	1.42	4,665						2.3	ND			
	13:30	3.7	0.58	1,048	28.3				4.68	0.03	ND	12	0.3	ND
	14:30	3.8	0.59	957	28.1				4.50	ND	ND	18	0.2	ND
	15:30		0.62	877	31.2				4.60	ND	ND	15	0.06	ND
	16:30	3.8	0.66	988	34.1				4.59	ND	ND	16	0.05	ND
	18:00	3.8	0.66	988	34.0				4.60	ND	ND	17	0.1	Trace
	19:00	3.7	0.71	1,039	36.2				4.59	ND	ND	15	0.1	Trace
5-15	9:55	1.5	4.33	3,703	14.2									
	10:10	1.6	1.89	4,892	69.0									
	10:20	2.5	1.02	4,085										
	10:35	3.0	0.64	3,007					4.35	0.8	ND	27	0.3	ND
	11:10	3.6	0.61	1,468					4.45	ND	ND	16	0.1	0.3



TABLE 12. RECYCLING WITH OXYGEN

Processing Rate: 16.7 cc/min  
 Oxygen Feed Rate: 0.2 l/min\*  
 Gas Recycle Rate: 6.8 l/min

Date	Time	Feed to Reactor			Vent Gas				Recovered Water					
		O <sub>2</sub> ,%	CO <sub>2</sub> ,%	N <sub>2</sub> O, ppm	CO <sub>2</sub> ,%	N <sub>2</sub> O, ppm	NH <sub>3</sub> , ppm	NO <sub>x</sub> , ppm	pH	NH <sub>3</sub> , mg/l	T.O.C., mg/l	Conduct, $\mu$ mhos	Turbidity, JTU	NO <sub>x</sub> , mg/l
5-15	12:50	14.4	3.0	4,870	12.2	ND	ND	ND	4.36	ND	ND	24	0.05	0.1
	13:25	14.4	2.8	4,840					4.40	ND	ND	23	0.05	
	14:10	14.8	2.7	3,630	11.7	ND	ND	ND						
	14:30	14.4	2.5	3,740	12.6	ND	ND	ND	4.40	ND	1.0	23	0.08	0.1
	15:30	13.9	3.0	3,850	13.2	ND	ND	ND	4.38	ND	ND	22	0.05	
5-16	11:20	1.5	4.5	23,340										
	11:30	7.1	4.7	22,770	18.1	ND	ND	ND						
	11:50	4.1												
	13:00		4.3	11,330					5.46	0.5	ND	55	0.1	
	13:50	16.7	4.4	11,220	18.4				4.71	0.9	ND	25	0.1	
	14:30	14.3	5.0	8,360	11.8				4.47	ND	ND	25	0.1	

\* Minimum O<sub>2</sub> feed rate possible with the present reactor design.

## CONCLUSIONS AND RECOMMENDATIONS

Based on test data obtained during this investigation, the following conclusions can be made:

1. Water recovered from untreated urine by the catalytic system meets the U.S. Drinking Water Standards, with the exception of a low pH.

2. The system can accept urine vapor produced by the evaporator of a vapor compression distillation unit at rates in excess of those required for a 3-man unit. Although the design processing rate was 0.86 l/hr, the highest observed rate was 1.2 l/hr.

3. The catalytic system can be operated either in a flow-through or a recycle mode. In both modes of operation, the oxygen required for the oxidation of  $\text{NH}_3$  and volatile organics can be supplied either as air or elemental oxygen to give 4-6% of  $\text{O}_2$  in the feed stream.

4. After startup, the system operates automatically and requires no adjustments or supervision, except supply of urine and removal of the recovered water.

5. The activity of the catalysts remained unchanged during the entire testing program consisting of daily runs and a duration test continued for 74 consecutive hours, adding to a total of 260 hours of operation.

The catalytic water recovery system is based on the treatment of urine vapor; therefore, its effective utilization will require integration with an energy efficient evaporative process capable of producing high urine vapor concentrations under spacecraft conditions. Although the catalytic system can accept vapor from a VCD unit, the current VCD systems

are packaged with evaporator-compressor-condenser inside of a shell which has no readily adaptable provisions for routing vapor to the catalytic  $\text{NH}_3$  oxidation reactor. In addition, the heat transfer and vapor condensation characteristics of the VCD system in presence of uncondensable gases are unknown. To make the catalytic water recovery system viable for spacecraft applications, it is recommended to conduct studies to determine the effect of noncondensibles and to develop methods for condensing water vapor produced by the catalytic system in a manner allowing the recapture of the latent heat of evaporation and its reutilization for urine evaporation under spacecraft conditions.